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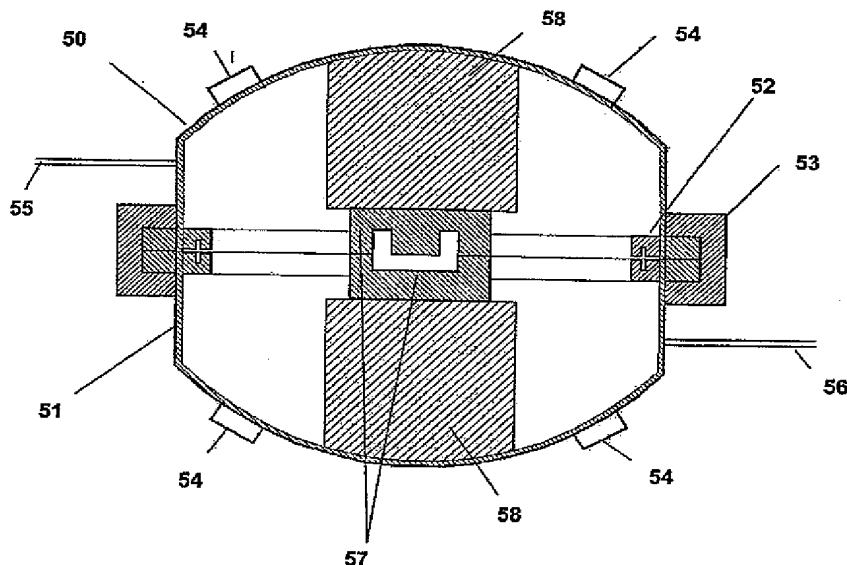
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[Continued on next page]

(54) Title: METHOD AND APPARATUS FOR PRODUCING BIODEGRADABLE FOAM



(57) Abstract: Methods, associated products and apparatus are described for the production of biodegradable foam products using a controlled pressure increase due to compressed air and a controlled pressure decrease in pressure as key variables during a microwave heating cycle to produce a foamed product. The biodegradable product formed has improved characteristics including a density from 10 to 100kg/m<sup>3</sup>; a soft and resilient structure; cushioning G-value characteristics to cushion an object with a fragility of 15 to 115; and a surface abrasion comparable to polystyrene.



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## METHOD AND APPARATUS FOR PRODUCING BIODEGRADABLE FOAM

### TECHNICAL FIELD

The present invention relates to a methods and associated products and apparatus  
5 for the production of biodegradable foam products. More specifically, the invention  
relates to methods and associated products and apparatus for the production of  
biodegradable foam products using pressure as a key variable to produce a  
product with improved characteristics including a low density.

### 10 BACKGROUND ART

The present invention builds on the inventions disclosed in WO 02/14043 and WO  
03/037598. In WO 02/14043 a two-stage microwave heating process is described  
for producing a biodegradable foamed product with improved packaging properties  
including resilience, compressibility and shock absorption. In WO 03/037598 a  
15 process and resulting foam is described for producing improved foam surface finish  
by causing the inner mould surface to heat to a predetermined temperature during  
processing and the use of a multiple magnetron microwave oven for heating.

Definitions used in these applications are included by reference herein.

The area of biodegradable packaging is widely discussed in the prior art. A variety  
20 of products and processing techniques exist that attempt to produce biodegradable  
foamed materials, as discussed in patent applications WO 02/14043 and WO  
03/037598.

Biopolymer packaging foams can be categorised as either: thin-walled moulded  
foams, suitable for applications such as containers, plates and cups; laminated and

agglomerated moulded foams, suitable for void filling and some shock absorption applications; thick-walled moulded foams, most suited to shock absorption applications.

A number of processes have been employed to produce thick-walled biopolymer foams suitable for packaging applications, including direct extrusion methods, 5 conductive heating methods, pressurised vessel methods, and volumetric heating methods such as microwave heating.

#### **Biodegradable moulded foam shapes produced by microwave heating**

This invention builds on the products and processes disclosed in WO 02/14043 10 (Blue Marble) and WO 03/037598 (Blue Marble). Such starch-based foam shapes formed by microwave heating have the attributes of up to one metre wall thickness and a smooth foam surface appearance achieved by elevating the mould wall temperature using a susceptor during processing. A multiple magnetron oven design is described. Pressure and rapid depressurisation means are contemplated 15 in passing with reference to WO 02/20238 (ATO). WO 03/037598 however, lacks enablement as it does not describe processing sequences using pressure, nor an apparatus design encompassing pressure and depressurisation functions. As discussed below WO 02/20238 also does not provide examples in any details for microwave processes that also encompass use of pressure.

20 WO 98/51466 (ATO) details a process for forming thick-walled biodegradable foam using a single step microwave heating process. The specification describes that it was important for the foaming process to proceed rapidly by either using a single microwave source having a high output or by a combination of microwave generator and mould, in which the pressure could be varied rapidly. No further 25 reference appears to be made towards pressure and as a result, this specification lacks enablement as no pressure ranges or heating sequences are described, nor

is there any description of a microwave and pressure capable apparatus. From the examples cited the best result is a 60 second microwave heating cycle time at atmospheric conditions which produces a foam product with a relatively high density of 150-160kg/m<sup>3</sup>.

- 5 EP 1347008 (Novamont) discloses a process for preparing foamed articles of biodegradable plastic material. Foaming particles prior to bonding, and bonding of particles utilising microwave heating are contemplated, however no detail of any apparatus or any heating profile is provided. Further, the examples do not describe microwave heating or use of pressure.

10 **Microwave oven designs for moulded foam shapes**

- US 4,908,486 (Nearctic) discloses a multiple magnetron microwave cavity designed for drying products. While disclosing the principles behind a multiple magnetron design to improve field uniformity, the apparatus is designed for drying applications and does not anticipate the issues associated with combining cavity or  
15 mould pressurisation / depressurisation in conjunction with rapid microwave heating, or of producing foamed articles.

- US 4,298,324 (Isobox-Barbier) discloses a microwave and mould design for expanding plastic resins. The cavity splits in half with one half remaining fixed and the other moving to allow ejection of the foam shape. Biodegradable resins are not  
20 contemplated, nor are multiple magnetron cavity designs, nor heating sequences incorporating elevated and reduced pressures.

**Combined Microwave and Pressure apparatus design**

Combined microwave pressure techniques are employed in a number of fields, including chemical digestion, sterilisation, sintering of metals and ceramics.

EP 0329338 (Alcan) discloses a process and apparatus for heating bodies to high temperatures at high pressures. The application is for sintering and isostatic pressing of ceramic powders to increase product density and does not contemplate biopolymer resins or a process of foaming to decrease product density, nor the use  
5 of microwave interactive moulds to form complex shapes.

Microwave autoclaves, such as that disclosed in US 5,436,432 (Cyr) have been employed for applications such as chemical digestion and analysis, and sterilization or retorting of food. Such devices do not contemplate the foaming of resins into complex shapes and the pressure/heating sequence required to achieve low  
10 density foams via such processes.

**Combined Microwave and Pressure apparatus design for moulded foam shapes**

WO 02/20238 (ATO) discloses a process for manufacturing thick-walled biodegradable foamed articles involving a rapid, discontinuous or semi-continuous  
15 process of subjecting a biopolymer to a heat and pressure increase by either injection of hot air or steam or alternatively, a heat increase only by use of microwave heating. A rapid depressurisation step is also considered in regard to hot air and steam methods i.e. by stopping flow of the hot air or steam (thus also stopping heating). The combination of microwave heating and pressure is not  
20 described. In addition, only one example is provided, utilising steam to achieve the elevated temperature and pressure profile, with a heating cycle time of 5 minutes. Such a long cycle time is not economic and therefore a process and apparatus capable of reducing the heating cycle time to less than one minute is more desirable. It is also the inventors' experience that, unless a very high quality steam  
25 is used (and hence higher cost), then use of steam can result in moisture forming on the exterior of the raw material, which, on foaming, causes surface faults on the

finished product that are undesirable for packaging applications.

A further application of pressurised microwave heating is for plastic foams as disclosed and discussed in DE 19654860 (Gefinex). Beads of unfoamed plastics are surface coated with a wetting agent and placed in a sealed mould. Microwave  
5 heating is used to generate steam as the blowing agent flashes off thus increasing the pressure in the mould and causing foaming as well as welding of the foamed particles together. Such applications do not contemplate the vapour dissipation issues associated with using water as the blowing agent nor the effect of vapour condensation on surface finish which is a key factor in regard to manufacture of  
10 biopolymer resins. Depressurisation rates in conjunction with elevated pressure and microwave heating are not described, nor are biodegradable resins.

WO 90/08642 (Adfoam) discloses an apparatus design and process for producing foamed plastic articles. The apparatus disclosed utilises 5kW magnetrons which are exponentially more expensive than standard domestic magnetrons and  
15 requires that the mould is moved within the cavity during processing to achieve a uniform microwave field. Depressurisation rates in conjunction with elevated pressure and microwave heating are not contemplated, nor are biodegradable resins.

According to the methods known in the art there is a trade-off between foam  
20 density, heating cycle time and apparatus cost. The prior art does not recognise the critical significance of the combination of the process parameters such as heat, pressure and depressurisation, and that without careful consideration, a low-density foam with an adequate surface finish cannot be produced in a cycle time of less than one minute.

25 Therefore there is a need for a method and apparatus that allows the production of a low density biodegradable foam product with adequate mechanical properties

that can operate within a heating cycle time of less than one minute.

It is an object of the present invention to address the foregoing problems or at least to provide the public with a useful choice.

All references, including any patents or patent applications cited in this specification  
5 are hereby incorporated by reference. No admission is made that any reference constitutes prior art. The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinency of the cited documents. It will be clearly understood that, although a number of prior art publications are referred to herein, this reference does not  
10 constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country.

It is acknowledged that the term 'comprise' may, under varying jurisdictions, be attributed with either an exclusive or an inclusive meaning. For the purpose of this specification, and unless otherwise noted, the term 'comprise' shall have an  
15 inclusive meaning - i.e. that it will be taken to mean an inclusion of not only the listed components it directly references, but also other non-specified components or elements. This rationale will also be used when the term 'comprised' or 'comprising' is used in relation to one or more steps in a method or process.

Further aspects and advantages of the present invention will become apparent  
20 from the ensuing description which is given by way of example only.

#### **DISCLOSURE OF INVENTION**

According to one aspect of the present invention, there is provided a method of producing a biodegradable foamed product including the steps of:



(a) placing a raw biodegradable material into a mould;

(b) locating the mould in a microwave cavity;

(c) conducting a microwave heating cycle;

characterised in that during step (c) the raw material is subjected to at least one  
5 controlled pressure increase and decrease using a compressed gas.

For the purposes of this invention, the term 'heating cycle' is defined as a time period that commences when the raw material for foaming is situated inside the closed mould and is ready for processing into a foam, and ends when the resultant foamed product is ready for removal from the mould.

10 It is the inventors' experience that unexpectedly, by controlling key parameters of the heating cycle including the pressure and microwave energy used, the level of expansion can be balanced against the level of shrinkage and hence a foam product can be produced at a reduced density and at a rapid speed.

The inventors have found that a fully moulded product with a continuous soft  
15 resilient foam surface is more easily achieved where pressure is increased during the heating cycle. Further, by using elevated pressure, the electric field strength that can be sustained without the occurrence of voltage breakdown (arcing and plasma formation) may be greatly increased and as a result, the heating time (and processing costs) may be reduced.

20 It is known that elevating pressure also elevates the boiling point of the blowing agent (water) resulting in greater vapour pressures being achieved inside the raw material before the water "flashes". This greater vapour pressure results in an increased magnitude of pressure drop experienced by the water vapour, which in turn increases expansion and therefore lowers the finished product foam density.

A yet further advantage of elevated pressure is that the melt viscosity of the raw material is reduced resulting in an increased expansion ratio and resultant lower foam density. If the melt viscosity is too low, the internal vapour pressure in the raw material may not be contained and the material easily ruptures. Conversely, it is the inventors' experience that there is an upper limit regarding the melt viscosity. If the melt viscosity is too high, the raw material may shrink excessively after initial expansion therefore causing an increase in foam density.

A further advantage of elevating pressure is that the rate of vapour loss by diffusion can be reduced. As a result more energy can be delivered to the starting material, without the loss of the blowing agent (water) or burning, resulting in an increase of vapour pressure within the raw material and therefore, reduced foam density. If water is the sole blowing agent, loss of vapour has a significant negative impact on both expansion and adhesion where starch-based pellets are used as the raw material.

Preferably, the compressed gas is air. More preferably, the gas or air source is not pre-treated such as by heating. This has the advantage that the pressurising agent is inexpensive and requires minimal pre-treatment.

In preferred embodiments, the raw biodegradable material is the substantially the same as that described in WO 02/14043 and WO 03/037598.

It is desirable that the starting materials are biodegradable and thus offer a significant environmental benefit over traditional materials such as polystyrene. This should not however be seen as limiting as it should be appreciated by those skilled in the art that other raw materials which have similar foaming characteristics to preferred materials may also be used in accordance with the present invention.

More particularly, the raw biodegradable material is derived from starch, cellulose,

protein or a derivative of starch, cellulose, or protein and combinations thereof.

Preferably, the raw material has a moisture content in the order of 5 to 30% wt with the moisture in the material acting as a blowing agent during the heating cycle.

Preferably, the raw biodegradable material is processed using a heat and shear  
5 generating process into pellets. Most preferably the process is extrusion.

Preferably, the heating cycle is completed in less than approximately 1 minute.

More preferably, the heating cycle is completed in the order of 30 seconds.

In preferred embodiments, the increased pressure is held for over half of the duration of step (c). By way of example, in one preferred embodiment, the  
10 increased pressure is held for approximately  $\frac{3}{4}$  of time to complete step (c).

It is the inventors' experience that, up to a point, the greater the time that the raw material is held under pressure, the greater the level of expansion. It is the inventors' understanding that if the time under pressure is too short, insufficient time occurs to allow initiation of boiling of the blowing agent (preferably water)  
15 contained in the pellets, and hence no or little expansion will result. This is thought to be because the raw material is not heated sufficiently to plasticize the material to a point where it flows and instead, the material ruptures immediately as a result of the expansion force of the internal vapour pressure when the pressure is reduced.

Preferably, the raw material is subjected to a pressure of between 1.5 and 100 bar  
20 during step (c). More preferably, the raw material is subjected to a pressure of between 3 and 20 bar during step (c). In preferred embodiments, the pressure is held at an approximately constant level although it should be appreciated that the pressure may be varied without departing from the scope of the invention for example, to optimise the pressure used to meet the characteristics of the raw  
25 material chosen.

It should be appreciated by those skilled in the art that the elevated pressure chosen will be in part a commercial decision. It is the inventors' understanding that the ideal pressure will at least in part be a compromise between improved characteristics obtained from the increased pressure levels against the increased cost of the pressure vessel design to account for greater pressures. It should be appreciated that capital costs increase substantially as greater extremes in pressure (or vacuum) are required.

Preferably, the pressure on the raw material is decreased in a controlled manner during step (c).

- 10 Preferably, microwave heating continues after the pressure is decreased in step (c). It is the inventors' experience that this additional heating improves the final foam product properties. Where processing time after the depressurisation is too short, significant shrinkage results, but conversely, if heating post depressurisation continues for too long, overcooking of the foam product may result.
- 15 It is understood by the inventors that if heating occurs for too long before initiating depressurisation, the internal vapour pressure inside the pellets become significantly greater than the pressure in the vessel resulting in vapour from the pellets escaping, drying the raw material out, and as a result leaving the material vulnerable to overcooking or drying.
- 20 A further unexpected finding by the inventors is that the rate of pressure decrease (depressurisation) may have a significant impact on the level of initial expansion and final foam density. It is the inventors' experience that when too high a rate of depressurisation occurs, an excellent initial expansion results but a high level of shrinkage post depressurisation also occurs. For a depressurisation rate that is too
- 25 slow, inadequate initial expansion results. The inventors have found that an optimum rate of depressurisation exists where the net effect of expansion and

shrinkage yields a foam product of the lowest density and the best surface appearance. This optimum rate may be achieved by matching the timing and rate of depressurisation to the temperature profile for a particular raw material where the temperature profile is a function of the vapour and melt temperature for a given pressure level. Given the above, a pressure decrease is initiated when the vapour and hence melt temperature is at its maximum before decomposition commences. For example, using the raw material used in the examples below, at 10bar pressure the maximum temperature is around 200°C and the rate of depressurisation is between 0.5-10 bar per second in order to produce the best foam.

10 Preferably, the pressure is reduced at a rate of 0.001 to 200 bar per second.

Preferably, the pressure is reduced rapidly at a rate of 0.5 to 10 bar per second during step (c). This range should not however be seen as limiting as it should be appreciated that the range is dependent on various factors such as the raw material used, the apparatus design, the heating rate achieved in the raw material and the initial increased pressure used.

15 In preferred embodiments, the pressure decreases within 0.1 to 10 seconds.

In a further preferred embodiment, the pressure decrease occurs as one continuous pressure drop although, it should be appreciated that more than one pressure decrease step may be used without departing from the scope of the invention.

20 It should also be realised that a key parameter in the pressure step is the pressure drop, hence, although a decrease to atmospheric conditions is a preferred embodiment, it may be beneficial to reduce the pressure to vacuum conditions.

Preferably, the pressure decrease commences in the last half of the overall time to complete step (c) although, it should be appreciated that this time may vary

depending on the raw material used as well as the pressure and heating profile used during the heating cycle.

In one alternative embodiment, the raw biodegradable material is subjected to a pressure increase before step (c) commences.

- 5 In another alternative embodiment, the raw biodegradable material is preheated before step (c) commences. Preferably, the raw biodegradable material may be preheated to a temperature below the raw biodegradable material melt temperature.

- 10 In one embodiment, the pressure increase and preheat steps described above undertaken before step (c), are completed at substantially the same time.

The preferred mould arrangement is substantially equivalent to embodiments encompassed and described in WO 03/037598. In particular, preferred mould embodiments of the present invention utilise a substantially microwave transparent mould material which is coated with a susceptor or microwave interactive material  
15 which causes the inner mould surface to heat during microwave heating.

Most preferably, the mould includes vents located on the mould walls. In preferred embodiments, vents are holes in the mould wall that are sized to be large enough to allow the dissipation of vapour from the blowing agent flashing off, but also small enough to achieve a smooth surface finish. Most preferably, the vent hole diameter  
20 is approximately 0.25 mm to 3 mm.

From the above discussion it should be appreciated that critical key processing factors have been identified to produce a biodegradable foamed product including:

- ❖ the pressure increase;
- ❖ the heating process;

- ❖ the timing of when the pressure is decreased (depressurisation); and,
- ❖ the rate of pressure decrease (depressurisation).

The method has the advantage of producing biodegradable foamed products with improved properties of a lower density, improved cushioning performance and improved surface finish, all being important characteristics in packaging applications. Further, the method may be completed rapidly therefore allowing greater production speed.

According to a further aspect of the present invention there is provided a biodegradable foamed product produced in accordance with the method substantially as described above.

According to a further aspect of the present invention there is provided a biodegradable foamed product with a thickness of up to approximately 1 metre manufactured from a biodegradable raw material with properties including:

- (a) a density from 10 to 100 kg/m<sup>3</sup>;
- (b) a soft and resilient structure;
- (c) cushioning G-value characteristics to cushion an object with a fragility of 15 to 115;
- (d) a surface abrasion comparable to polystyrene.

Preferably, the density is from 20 to 60 kg/m<sup>3</sup>.

It is understood by the inventors that the cushioning performance is effected at least in part by incomplete expansion in a mould, and the resilience of the surface of the foam. It is the inventors' experience that an ideal foamed product may be

obtained by a manufacturing method that minimises the number of voids within the foam.

Cushioning factor may be measured by a number of techniques including that outlined in British Standard BS7539. In order to protect articles with a fragility factor of between 15 and 115 it is desirable for the foam to have a G-value in a similar range. G-value for a foam is understood to give a foam with the ability to sufficiently attenuate shock and vibrations such that the packaged article under normal circumstances is unlikely to be exposed to a G-force greater than this numerical value. The higher the foam G-value the less suitable it might be for packaging fragile or delicate articles. The product of the present invention has a G-value to adequately protect items with a fragility factor between 15 and 115.

Surface abrasion characteristics can be described by way of an abrasion test. In tests carried out on foam samples of the present invention, abrasion was tested by rubbing foam across aluminium sheets of differing hardness values. No difference in abrasion level was found by the inventors between samples of expanded polystyrene and the product of the present invention when samples were compared using the abrasion method described.

It should be appreciated from the above description of the product that the product of the present invention has significantly improved characteristics over the prior art. In particular, the reduced density of the foam product in combination with improved cushioning characteristics means that the product is ideally suited for packaging applications. Given that the foam product is biodegradable, the invention provides an environmentally friendly alternative to polystyrene which is used in most packaging applications.

According to a further aspect of the present invention there is provided an apparatus for the production of a foamed product with a thickness of up to



approximately 1 metre including:

- (a) a cavity;
  - (b) a mould capable of containing a raw material that is able to be melt processed when subjected to heat and pressure treatment to form a foam;
  - (c) at least one magnetron capable of microwave heating the raw material in a microwave heating cycle;
  - (d) at least one inlet through which a compressed gas passes; and,
  - (e) at least one outlet for depressurisation;
- 10 characterised in that the apparatus is capable of subjecting the raw material to controlled pressure increases and decreases using compressed gas in conjunction with microwave heating.

Preferably, the compressed gas is air although it should be appreciated that a wide variety of gases may be used to place the raw material under pressure without departing from the scope of the invention.

In one embodiment, the microwave cavity including the mould and raw material is pressurised. In an alternative embodiment the apparatus further includes a sealed chamber which acts the pressure vessel (hereafter referred to as the chamber) within which the mould and raw material are placed, the chamber is positioned inside the apparatus cavity, and the chamber containing the mould and raw material, is pressurised.

Preferably, the raw material is a biodegradable raw material. It should be appreciated that other materials may also be used in accordance with the present

invention and this should not be seen as limiting. For example, other non-biodegradable raw materials may also be used such as plastics and polystyrene precursors.

Preferably, the apparatus is designed to handle a pressure of between 1.5 and 100  
5 bar. More preferably, the pressure is between 3 and 20 bar.

Preferably, the apparatus is designed to be able to reduce the pressure during microwave heating. More preferably, the apparatus is designed to enable a pressure decrease at a rate of 0.001 to 200 bar per second.

Preferably, the apparatus includes a plurality of magnetrons, the exact number  
10 depending on factors such as the size of the cavity. Preferably, the magnetrons are capable of heating the raw material at a rate of up to 25°C per second. More preferably, the rate of heating is between 2°C and 10°C per second.

Preferably, the magnetrons operate at a frequency from approximately 915 MHz to  
5 GHz. Most preferably, the frequency is an approximately constant level of 2450  
15 MHz (domestic microwave frequency). It should be appreciated that use of domestic microwave frequencies is preferable to reduce the need for manufacture of tailored magnetrons for the apparatus and therefore minimising capital costs.

As described in WO 03/037598, by utilising a plurality of magnetrons situated around the walls of the cavity, a uniform field can be established within a  
20 pressurised cavity. This has the advantage of avoiding the need for mode stirrers or movement of the raw material during the heating cycle.

Preferably, the apparatus includes at least one inlet for pressurising the cavity or vessel if used. It should be appreciated that where multiple inlet ports are used, the time taken to pressurise the cavity or vessel to the desired pressure level can  
25 be minimised. A design consideration also is that if ports are used that are too

large in size, they may interfere with the microwave feed arrangement hence a number of smaller inlet ports is preferable.

Preferably, the apparatus includes at least one outlet for depressurising the cavity or pressure vessel if used. In preferred embodiments, multiple outlet ports may be  
5 located around the microwave cavity to enable depressurisation to be effected. A plurality of ports may be used to vary the rate of depressurisation. Alternatively, the rate of depressurisation may be varied using a flow-restricting device. Preferably, the outlet is a valve.

Preferably, the cavity shape is selected from group consisting of: cylindrical,  
10 asymmetrical hexagonal or semi-elliptical. Most preferably, it is the inventors' experience that the cavity size should be a multiple of the microwave frequency wavelength.

Preferably, apparatus includes at least one wave guide. Preferably, the number of wave guides used match the number of magnetrons used although, this should not  
15 be seen as limiting as it should be appreciated that additional wave guides may be used as may be required for design purposes. Preferably, wave-guides connect the cavity to the power modules used to generate microwaves via wave-guide ports.

Preferably, the apparatus includes at least one pressure window located between  
20 the waveguide exit point and the cavity. Preferably, the pressure window or windows are manufactured from a substantially microwave transparent material such as quartz or Teflon™. In preferred embodiments, the window also includes a thin sacrificial window made from mica. A sacrificial window may be useful as, should any arcing or plasma occur, the relatively cheaper sacrificial window will be  
25 damaged therefore protecting the more expensive pressure window.

Preferably, the apparatus includes a pressure relief valve. In preferred embodiments, the pressure relief valve is piped to a spacing (preferably encapsulated with air) between two pressure windows. If the pressure in this space exceeds a pre-determined level the relief valve opens and vents gas.

- 5 Alternatively, the apparatus may include microwave launchers in place of the waveguides to feed microwave energy directly to the cavity from the magnetrons.

Preferably, the cavity may be opened and closed to allow insertion and removal of the mould and raw material. More preferably, the cavity splits apart.

- 10 In an alternative embodiment, the cavity, mould and chamber if used, have an injection point through which, by use of an injection gun, raw material can be inserted into the mould. Preferably, the injector gun or guns are connected to at least one feed hopper outside the microwave cavity. The injector guns also house ejector pins. After loading of the pellets, ejector pins are positioned flush with the inner mould surface to plug the mould ports. Ejection of the foam is achieved when  
15 the ejector pins are pushed proud of the inner mould surface. Additional ejector pins may be located in the lower half of the mould.

- Preferably, a ring or hoop mechanism is used to seal the cavity and pressure chamber if used. In one preferred embodiment, the apparatus is formed from two halves, each half having a castellated flange. A castellated locking ring is rotated  
20 or clamped to seal to the apparatus. Chokes may also be added to the inner cavity surface where the cavity halves join to eliminate microwave leakage.

Preferably, interlock devices are also included as part of the sealing process to ensure that the apparatus cannot be operated without adequate sealing of the cavity.

In preferred embodiments, structures such as microwave transparent platens are included inside the cavity or pressure vessel if used to house the mould arrangement. Such structures may be mounted in each of the cavity halves.

Once the heating cycle is completed using the apparatus, the product may be  
5 removed from the apparatus or at least the mould. In one embodiment, the product is removed using compressed air forced through the mould vents.

The preferred mould arrangement is substantially equivalent to embodiments encompassed and described in WO 03/037598. In particular, preferred mould  
embodiments of the present invention utilise a largely microwave transparent  
10 mould material which is coated with a susceptor or microwave interactive material which causes the inner mould surface to heat during microwave heating.

One advantage of the above described apparatus is the size of foamed product that may be produced. Prior art methods such as autoclaves and selected microwave pressure vessels exist for uses such as chemical analysis however,  
15 only very small sample sizes can be processed. The apparatus of the present invention that includes multiple magnetrons, pressure and microwave leakage seals, as described above, allow for the production of biodegradable foam products up to one metre thick.

A further advantage of the apparatus of the present invention is that the apparatus  
20 is comparatively inexpensive to manufacture and operate when compared with existing apparatus. A yet further advantage is that, due to the significantly reduced cycle time, processing speeds are greatly increased compared to prior art methods.

The apparatus of the present invention also has the advantage of producing a product with significantly improved properties over prior art methods including but  
25 not limited to a reduced density. This is of particular importance for packaging

applications.

### **BRIEF DESCRIPTION OF DRAWINGS**

Further aspects of the present invention will become apparent from the ensuing  
5 description which is given by way of example only and with reference to the  
accompanying drawings in which:

Figure 1 is a cross-section elevation view of one preferred pressure vessel  
embodiment of the present invention where a chamber is used  
located within a cavity;

10 Figure 2 is a cross-section elevation view of a further embodiment of the  
present invention where the microwave cavity is pressurised; and,

Figure 3 is a cross-section elevation view of a pressure window for a wave-  
guide port used in the embodiment of figure 2.

### **BEST MODES FOR CARRYING OUT THE INVENTION**

The apparatus, method of operation and product are now disclosed with reference  
to the embodiments shown in figures 1 to 3.

#### **Apparatus**

One embodiment for the apparatus of the present invention is shown in Figure 1.

20 The apparatus consists of a microwave cavity 1 that has multiple magnetrons (not  
shown). The cavity 1 includes multiple microwave wave-guide ports 9 situated  
throughout the cavity 1 walls. A mould 3 with vents (not shown) filled with raw

material (not shown) is located within a pressure vessel 2 manufactured from microwave transparent material. The mould 3, raw material and pressure vessel 2 are all located within the microwave cavity 1. The pressure in the pressure vessel 2 is increased by inserting compressed air through inlet valve 8. The pressure  
5 vessel 2 is depressurised via a flow regulated valve 6. Air released from the pressure vessel 2 via valve 6 is vented into a chamber 7. The prime aim of this chamber 7 is to dissipate noise and retain air released. A safety relief valve 5 vents if the pressure in the pressure vessel 2 exceeds the maximum predetermined pressure. A choke system 4 is situated at the junction of the pressure vessel 2 and  
10 the microwave cavity 1. This is included to prevent leakage of microwaves from the cavity 1 during a heating cycle.

A second alternative embodiment is shown in Figure 2.

The apparatus consists of a microwave cavity made up of two halves 50,51 which, when assembled, forms a pressure vessel. A microwave choke system 52 is  
15 included to prevent leakage of microwaves from the cavity halves 50,51. A locking mechanism 53 clamps the cavity halves 50,51 together when in use. Mould halves 57 are clamped together at the same time as the two cavity halves 50,51 are clamped together. Locator pins (not shown) ensure the mould halves 57 and cavity halves 50,51 are aligned correctly. Microwave wave-guide ports 54 are situated  
20 around the cavity halves 50,51 and are located to minimise cross-coupling of microwaves during operation. An inlet port 55 is located on one side of the cavity 50. Compressed air or other gases are inserted into the cavity via the inlet port to pressurise the cavity. The cavity 50,51 is depressurised via an outlet port 56. Air or gas released from the cavity 50,51 via outlet port 56 is vented into a chamber  
25 (not shown). Support structures 58 brace the mould against the cavity walls 50,51.

Figure 3 shows a close up view of wave-guide port 54 shown in Figure 2. The

wave-guide port 54 has a sacrificial window 100 at the interface of the cavity 50,51 wall through which microwaves enter the cavity 50,51. Behind the sacrificial window 100 are further pressure windows 101 separated by an air pocket 102 with a safety relief valve 103. The valve 103 vents if the pressure in the cavity 50,51 reaches unsafe levels, thus protecting the microwave heating modules (not shown) attached to the wave-guides located in the guide port 54.

## Methods of Manufacture

### Method Example 1

This example shows a best method for production of a foamed product based on trials completed by the inventors.

1. Place 110 grams of starch pellets (raw material) formed by extrusion and containing approximately 22% wt moisture into a mould 3, 57 manufactured from ULTEM polyetherimide. The inner mould 3, 57 surfaces are coated with a ferrite/silicone rubber liner (not shown) which acts as a susceptor or microwave interactive material that heats during the heating cycle. The mould 3,57 shape is cylindrical with a diameter of 105mm, and a length of 255mm. The mould 3,57 is vented on all walls. More information regarding preferred moulds and configurations 3,57 is discussed in PCT/NZ/0200226.
2. Clamp the mould 3,57 shut.
3. Place the mould 3,57 inside an apparatus as described above and shown in figures 1 to 3;
4. Seal the chamber 2 if used, and cavity 1,50,51;
5. Set the power level to a maximum power of 16 magnetrons arranged around the microwave cavity 1,50,51;



6. Increase the pressure in the cavity 50,51 or chamber 2 (and mould 3,57 contained within) to 10 bar over a period of 5 seconds;
7. Commence the heating cycle with the pressure held at approximately 10 bar for a time period of 22 seconds;
- 5 8. After 22 seconds, depressurise the cavity 50,51 or chamber 2 to atmospheric pressure via a ½ inch valve 6 or outlet port 56, taking approximately 5 seconds to return the cavity 50,51 or chamber 2 to atmospheric pressure;
9. After approximately 3 more seconds the heating cycle is stopped and the foamed product removed.
- 10 The product formed from the above process is a fully formed foam shape with a density in the order of 42kg/m<sup>3</sup> and a smooth and resilient foam surface.

The surface has an abrasion level and cushioning characteristics directly comparable to polystyrene.

#### **Method Example 2**

- 15 The effect of a two stage depressurisation method is shown below with variations in the first pressure used.

The procedure used was as follows:

1. The power level was set to 16 magnetrons;
2. 110g of the raw material of method example 1 was placed inside the cylindrical  
20 mould 3 of method example 1;
3. The mould 3 was placed inside the chamber 2 and located inside the microwave cavity 1

4. 10 bar pressure was applied in the chamber 2;
5. The raw material was then heated for 22 seconds after which time the 1/2" depressurisation valve 6 was opened to allow the pressure to drop to 1.5 bar;
6. After 30 seconds total cycle time (including the 22 seconds of step 5) a second 4" decompression valve 6 was opened to allow the pressure in the chamber 2 to lower to atmospheric pressure;
7. The mould 3 was then removed from the chamber 2 and the foam product removed;
8. Steps 1 to 7 were then repeated for initial applied pressures of 8, 6, 4, 2, 1.5, and 0 bar.

In summary, the pressure profiles used were as follows:

Time	Pressure Profile Number						
	1	2	3	4	5	6	7
0	10	8	6	4	2	1.5	0
22	1.5	1.5	1.5	1.5	1.5	1.5	0
30	0	0	0	0	0	0	0

This trial showed that the greater the initial pressure level, and hence the pressure difference, the fuller the foam shape.

### 15 Method Example 3

The effect of a two stage depressurisation method is shown below with variations in the second pressure used.

1. The power level was set to 16 magnetrons;

2. 110g of the raw material of method example 1 was placed inside the cylindrical mould 3 of method example 1;
3. The mould 3 was placed inside the chamber 2 and located inside the microwave cavity 1;
- 5 4. 10 bar pressure was applied in the chamber 2;
5. The raw material was then heated for 22 seconds after which time the 1/2" depressurisation valve 6 was opened to allow the pressure to drop to 8.0 bar;
6. After 30 seconds total cycle time (including the 22 seconds of step 5) a second 4" decompression valve 6 was opened to allow the pressure in the chamber 2 to lower to atmospheric pressure;
- 10 7. The mould 3 was then removed from the chamber 2 and the foam product removed;
8. Steps 1 to 7 were then repeated for initial applied pressures of 6, 4, 2, 1.5, 0 bar.
- 15 In summary, the pressure profiles used were as follows:

Time	Pressure Profile Number						
	1	2	3	4	5	6	7
0	10	10	10	10	10	10	10
22	8	6	4	2	1.5	0	0
30	0	0	0	0	0	0	0

The results found were as follows:

<i>Time</i>	<b>0</b>	<b>22</b>	<b>30</b>	<i>Comments</i>
Profile 1	10	8	0	Excellent expansion as seen by flashing and vent hole marks but some shrinkage.
Profile 2	10	6	0	Excellent expansion as seen by flashing and vent hole marks but some shrinkage, although not to the extent of profile 1
Profile 3	10	4	0	Equal worst sample in terms of filling Poor filling in comparison to 10-0-0 bar $\Delta P$
Profile 4	10	2	0	Equal worst sample in terms of filling Poor filling in comparison to 10-0-0 bar $\Delta P$
Profile 5	10	1.5	0	Good expansion, low shrinkage, similar to profile 6
Profile 6	10	0	0	The best of all samples in this set Good expansion, low shrinkage so net effect was best density

In summary, the best net result (considering expansion and shrinkage) was achieved with a large initial and small final pressure drop i.e. 10-1.5-0 bar and 10-0-0 bar.

#### **Method Example 4**

A one step depressurisation step method is shown below with variations in the timing of the depressurisation step shown.

1. The power level was set to 16 magnetrons;
2. 110g of the raw material of method example 1 was placed inside the cylindrical mould 3 of method example 1;

3. The mould 3 was placed inside the chamber 2 and located inside the microwave cavity 1;
  4. 10 bar pressure was applied in the chamber 2;
  5. The raw material was then heated for 8 seconds after which time the 4" depressurisation valve 6 was opened to allow the pressure to drop to 0 bar;
  6. After 30 seconds total cycle time (including the 8 seconds of step 5) the mould 3 was then removed from the chamber 2 and the foam product removed;
  7. Steps 1 to 6 were then repeated for pressurisation times of 14, 18, 22, 26 seconds.
- 10 In summary, the pressure profiles used were as follows:

Time	Pressure Profile Number				
	1	2	3	4	5
0	10	10	10	10	10
4	10	10	10	10	10
8	0	10	10	10	10
14	0	0	0	10	10
18	0	0	0	0	10
22	0	0	0	0	10
26	0	0	0	0	0
30	0	0	0	0	0

The results found were as follows:

- For pressure drops at times 14, 18 & 22 seconds (profiles 2 to 5), it was found that the greater the time until the pressure drop, the greater the level of expansion. For profile 1 where the pressure drop was undertaken after 8 seconds, the expansion was poor with poor adhesion in the final product. This result is most likely because 8 seconds was insufficient time to have initiated 'boiling' of the water (blowing

agent) hence no expansion resulted from the large pressure drop.

The sample produced using profile 4 (14 seconds) showed signs of the pelleted raw material having ruptured at the time of pressure drop. This is most likely the result of subjecting the pellets to explosive decompression before the pellets had  
5 heated sufficiently to 'plasticize' the pellets to a point where they would flow rather than rupturing as the result of the expansion force of the internal vapour pressure.

For the sample produced using profile 5 (26 seconds), the expansion was good with flashing indicating high pressures were reached, but shrinkage appeared to be a problem with the sample produced as the sample had smaller dimensions  
10 than the sample produced using profile 4.

#### **Method Example 5**

A one step depressurisation process trial was undertaken to determine whether or not increasing processing time before pressure drop aids in foam expansion.

As the processing time before the pressure drop was increased from 22 seconds to  
15 34 seconds and 38 seconds, the level of foam expansion decreased. The raw material pellets also showed signs of overcooking as the processing time before the pressure drop increased.

It is thought that at processing times (before the pressure drop) of over 22 seconds, the internal vapour pressure inside the pellets was significantly greater than the  
20 pressure in vessel resulting in blowing agent (water) escaping the raw material before expansion. As a result that material dried out and became overcooked.

#### **Method Example 6**

A one step depressurisation process is shown below with variations used in the amount of time that microwave heating continues after the depressurisation step.

The object of this trial was to assess whether or not increasing the processing time after the pressure drop could aid in minimising shrinkage of the foam.

It was found that as the processing time after the pressure drop increased, the amount of shrinkage decreased. However, at post depressurisation processing times greater than 8 seconds, the foam samples showed significant areas of overcooking.

### Method Example 7

Trials are shown below testing the effect that the rate of depressurisation has on the product produced.

- 10 For each experiment with differing depressurisation rates, different start pressures were also used to determine if the start pressure also had any effect.

The following procedure was undertaken for each of the samples:

1. The same raw material of method example 1 was placed inside a cylindrical mould 3 with characteristics as per step 1 of method example 1.
- 15 2. The loaded mould 3 was placed inside a chamber 2.
3. Pressures of 1.5, 2.5, 5.0, 7.5 and 10 bar were applied to the chamber 2 for each trial respectively.
4. The heating cycle was then started using a total of 10 magnetrons and a processing time of 40 seconds for all trials.
- 20 5. The pressure in the cavity 2 was then released in one continuous step immediately after the heating cycle had completed using three different sized depressurisation valves 6 (large sized (4" butterfly), intermediate sized (1/2"

ball) and small sized (1/4" ball)) which allowed differing rates of depressurisation.

Results found from the above trials concluded that:

- ❖ The higher the pressure used, the greater the degree of overcooking.
- 5      ❖ At all the pressure levels investigated, when depressurising at the end of the cycle, the higher the rate of depressurisation, the higher the level of expansion achieved.

More specifically:

- ❖ When depressurising through the largest valve 6 at the end of the heating cycle, the higher the 'applied' pressure, the greater the 'initial' expansion but also, the greater the shrinkage observed.
- 10      ❖ When depressurising through the intermediate sized valve 6 at the end of the heating cycle, the higher the 'applied' pressure, the lower the expansion achieved.
- 15      ❖ When depressurising through the smallest valve 6 at the end of the heating cycle, the higher the 'applied' pressure, the lower the expansion achieved.

It should be appreciated from the above examples that there are provided methods and associated products and apparatus to produce a foamed product with improved foaming properties to result in a product with a low density, improved cushioning characteristics and a surface finish and abrasion level comparable to polystyrene.

20

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto



without departing from the scope thereof as defined in the appended claims.

**WHAT WE CLAIM IS:**

1. A method of producing a biodegradable foamed product including the steps of:

(a) placing a raw biodegradable material into a mould;

(b) locating the mould in a microwave cavity;

(c) conducting a microwave heating cycle;

characterised in that during step (c) the raw material is subjected to at least one controlled pressure increase and decrease using a compressed gas.

2. The method of claim 1 wherein the compressed gas is air.

3. The method of either claim 1 or claim 2 wherein the compressed gas is not pre-treated.

4. The method as claimed in any of the above claims wherein the raw biodegradable material is derived from starch, cellulose, protein or a derivative of starch, cellulose, or protein and combinations thereof.

5. The method as claimed in any of the above claims wherein the raw material has a moisture content in the order of 15 to 50% wt.

6. The method as claimed in any of the above claims wherein the raw biodegradable material is pre-formed by a heat and shear process into pellets.

7. The method as claimed in any of the above claims wherein step (c) is completed in under one minute.

8. The method as claimed in any of claims 1 to 6 wherein step (c) is completed

in the order of 30 seconds.

9. The method as claimed in any of the above claims wherein the increased pressure is maintained for over half of the duration of step (c).

10. The method as claimed in any of the above claims wherein the raw material is subjected to a pressure of between 1.5 and 100 bar during step (c).

11. The method as claimed in any of claims 1 to 9 wherein the raw material is subjected to a pressure of between 3 and 20 bar during step (c).

12. The method as claimed in any of the above claims wherein microwave heating continues after the pressure is decreased during step (c).

13. The method as claimed in any of the above claims wherein the pressure decrease occurs at a rate of 0.001 to 200 bar per second.

14. The method as claimed in any of the above claims wherein the pressure is decreased rapidly at a rate of 0.5 to 10 bar per second during step (c).

15. The method as claimed in any of the above claims wherein the timing and rate of pressure decrease is matched to the temperature profile for the raw material.

16. The method as claimed in any of the above claims wherein the pressure decreases within 0.1 to 10 seconds.

17. The method as claimed in any of the above claims wherein the pressure decrease occurs as one continuous pressure drop.

18. The method as claimed in any of the above claims wherein the pressure decrease commences in the last half of the overall time to complete step (c).

19. The method as claimed in any of the above claims wherein the raw material is subjected to a pressure increase before step (c) commences.
20. The method as claimed in any of the above claims wherein the raw material is preheated before step (c) commences.
21. The method as claimed in claim 20 wherein the raw biodegradable material is preheated to a temperature below the raw biodegradable material melt temperature.
22. The method as claimed in any of the above claims wherein the mould is substantially microwave transparent and is coated with a susceptor material.
23. The method as claimed in any of the above claims wherein the mould includes vents.
24. A biodegradable foamed product produced in accordance with the method as claimed in any of claims 1 to 23.
25. A biodegradable foamed product with a thickness of up to approximately 1 metre manufactured from a biodegradable raw material with properties including:
- (a) a density from 10 to 100 kg/m<sup>3</sup>;
  - (b) a soft and resilient structure;
  - (c) cushioning G-value characteristics to cushion an object with a fragility of 15 to 115;
  - (d) a surface abrasion comparable to polystyrene.
26. An apparatus for the production of a foamed product with a thickness of up to approximately 1 metre including:

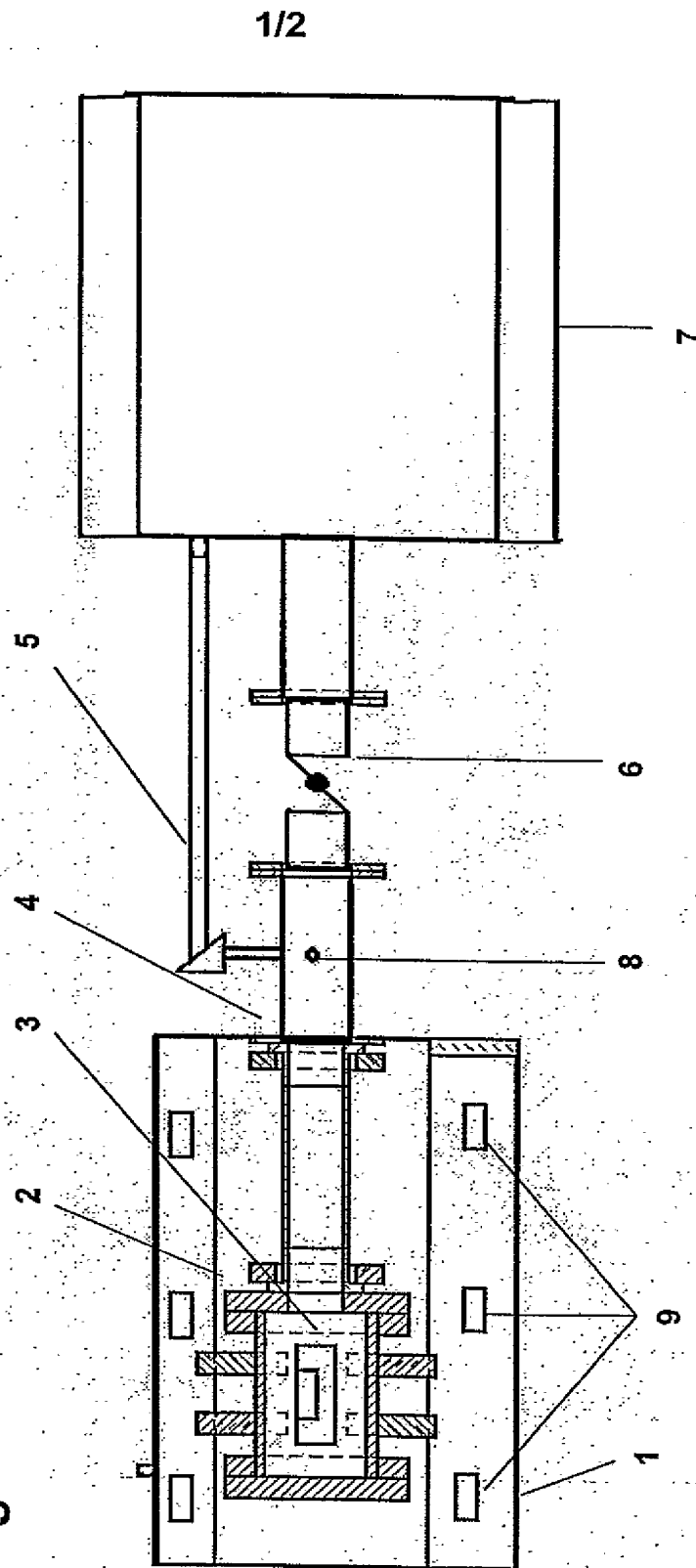
- (a) a cavity;
- (b) a mould capable of containing a raw material that is able to be melt processed when subjected to heat and pressure treatment to form a foam;
- (c) at least one magnetron capable of microwave heating the raw material in a microwave heating cycle;
- (d) at least one inlet through which a compressed gas passes; and,
- (e) at least one outlet for depressurisation;

characterised in that the apparatus is capable of subjecting the raw material to controlled pressure increases and decreases using compressed gas in conjunction with microwave heating.

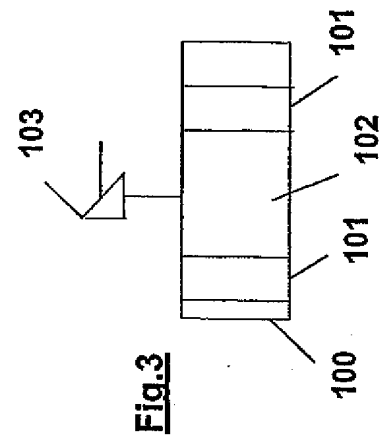
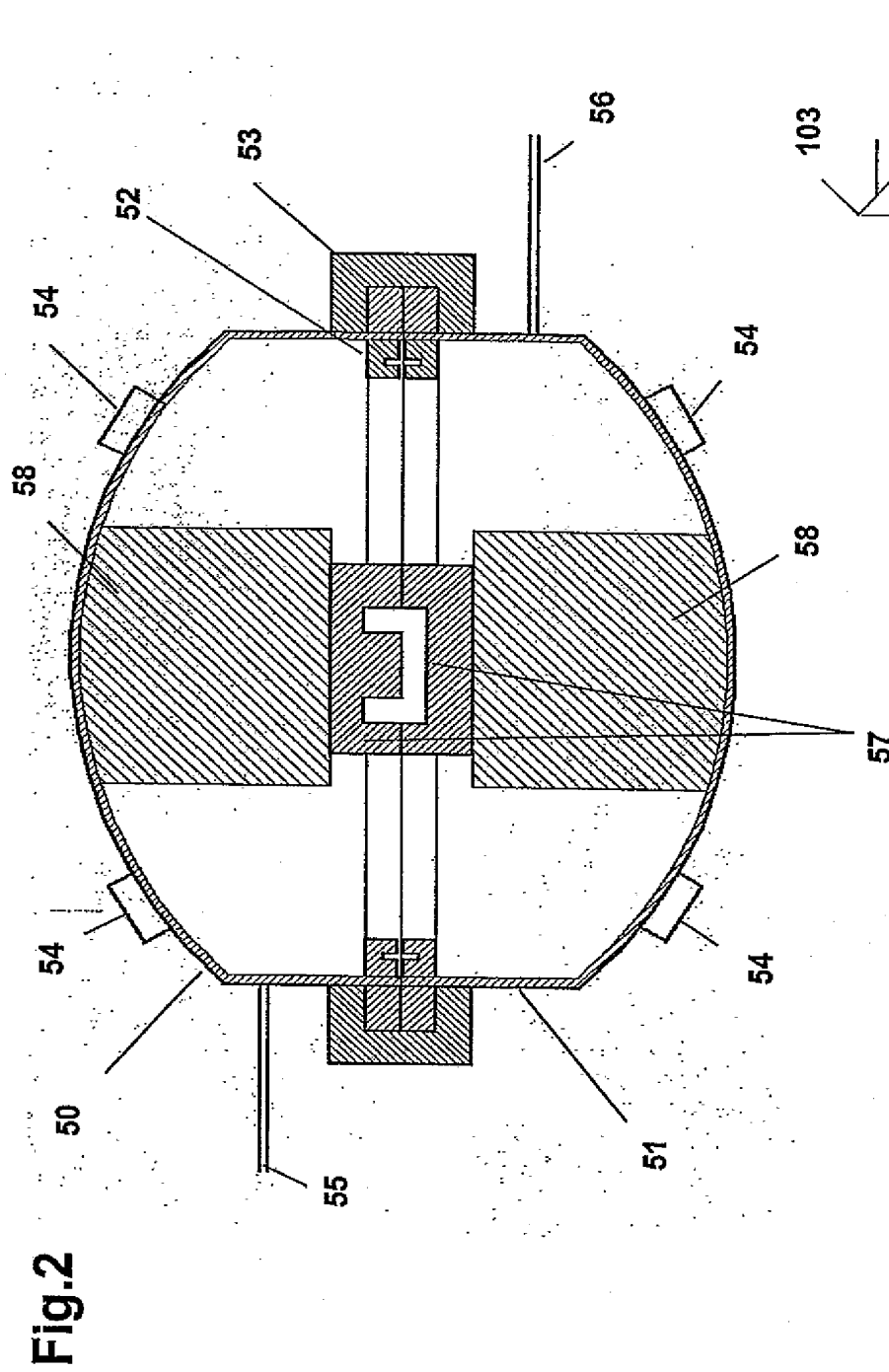
- 27. The apparatus as claimed in claim 26 wherein the compressed gas is air.
- 28. The apparatus as claimed in either claim 26 or claim 27 wherein the apparatus further includes a sealed chamber within which the mould and raw material are placed, the chamber is positioned inside the apparatus cavity, and the chamber containing the mould and raw material, is pressurised.
- 29. The apparatus as claimed in any of claims 26 to 28 wherein the outlet is a valve.
- 30. The apparatus as claimed in any of claims 26 to 29 wherein the magnetrons are capable of heating the raw material at a rate of up to 25°C per second.
- 31. The apparatus as claimed in any of claims 26 to 30 wherein the magnetrons operate at a frequency from approximately 915 MHz to 5 GHz.

32. The apparatus as claimed in any of claims 26 to 30 wherein the magnetrons operate at a frequency of an approximately constant level of 2450 MHz.
33. The apparatus as claimed in any of claims 26 to 32 wherein the apparatus includes at least one pressure window manufactured from a substantially microwave transparent material and located between a waveguide exit point and the cavity.
34. The apparatus as claimed in claim 33 wherein the window includes a sacrificial window.
35. The apparatus as claimed in any of claims 26 to 34 wherein the apparatus includes an injection point through which raw material can be inserted into the mould.
36. The apparatus as claimed in any of claims 26 to 35 wherein the mould is substantially microwave transparent and is coated with a susceptor material.

Fig.1



2/2





# INTERNATIONAL SEARCH REPORT

International application No.

PCT/NZ2004/000305

## A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl. <sup>7</sup>: B29C 35/08

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DWPI: B29C 35/08, 35/12, 44/+, 67/20 (IPC 7); B29C 67/22 (IPC 5); B29D 27/+ (IPC 3) & Keywords - foam+, compress+, +pressur+, microwav+, dielectric+, biodegrad+, cellulose+, starch+, protein+, thickness, polystyrene

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2003/037598 A (BLUE MARBLE POLYMERS LIMITED) 8 May 2003 Whole document	25
A	WO 2002/020238 A (ATO B.V.) 14 March 2002 Whole document	1-36
A	WO 1990/008642 A (ADFOAM PTY. LIMITED) 9 August 1990 Whole document	1-36
A	EP 0329 338 A (ALCAN INTERNATIONAL LIMITED) 23 August 1989 Whole document	1-36

☒ Further documents are listed in the continuation of Box C

☒ See patent family annex

* Special categories of cited documents:	
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
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Date of the actual completion of the international search 22 February 2005	Date of mailing of the international search report 01 MAR 2005
Name and mailing address of the ISA/AU AUSTRALIAN PATENT OFFICE PO BOX 200, WODEN ACT 2606, AUSTRALIA E-mail address: pct@ipaustalia.gov.au Facsimile No. (02) 6285 3929	Authorized officer  JACOB ELIJAH Telephone No : (02) 6283 2161

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/NZ2004/000305

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member	
WO 03037598	CA 2463921	EP 1453648	NZ 515097
WO 0220238	AU 96087/01		
WO 9008642	NONE		
EP 0329338	US 5010220		
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.			
END OF ANNEX			